**Designation: ~~D5671 – 95 (Reapproved 2019)~~ D5671 – 20**

# Standard Practice for Polishing and Etching Coal Samples for Microscopical Analysis by Reflected Light[1]

This standard is issued under the fixed designation D5671; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers laboratory procedures for preparing an etched, polished surface of granular and block samples of coal for examination with a microscope using reflected light illumination.

1.2 *Units*—The values stated in SI units are to be regarded as standard. ~~No other units of measurement are included in this~~ The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*



## 2. Referenced Documents

2.1 *ASTM Standards:*[2]



D121 Terminology of Coal and Coke
D2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light
D2798 Test Method for Microscopical Determination of the Vitrinite Reflectance of Coal
D2799 Test Method for Microscopical Determination of the Maceral Composition of Coal
D4596 Practice for Collection of Channel Samples of Coal in a Mine
D5192 Practice for Collection of Coal Samples from Core

## 3. Terminology

3.1 Terminology used in this standard can be found in Terminology D121.

## 4. Summary of Practice

4.1 A subsplit of a representative sample obtained in accordance with Practice D4596 and prepared in accordance with Practice

[1] This practice is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.28 on Petrographic Analysis of Coal and Coke.

Current edition approved ~~Nov. 1, 2019~~Oct. 1, 2020. Published ~~November 2019~~November 2020. Originally approved in 1995. Last previous edition approved in ~~2011~~2019 as D5671~~—95(2011).~~ – 95(2019). DOI: ~~10.1520/D5671-95R19.~~10.1520/D5671-20.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D2797, or a block of coal obtained in accordance with Practice D5192, is polished to a flat, scratch-free ~~surface,~~surface; the reflectance of vitrinite is determined on a subsplit in accordance with Test Method D2798~~;~~; and then other subsplits are chemically etched using an acidified potassium permanganate solution.

## 5. Significance and Use

5.1 Components observable in surfaces of coal samples prepared in accordance with the laboratory procedures of this practice will have differential relief that will aid in their maceral identification by visual classification and enables identification of plant parts or tissues that formed the coal.

5.2 Samples prepared by this practice can be used for microscopical determination of the volume percent of physical components of coal in accordance with Test Method D2799.

5.3 Samples prepared by this practice can be used for the microscopical identification of botanical components by taphonomic rank. Samples for this purpose should be limited to telovitrinite and semifusinite rich coals of bright lithotype to maximize feedback from etching.

## 6. Apparatus

6.1 *Ultrasonic ~~Cleaner—~~Cleaner,* large enough for sample holder and sample preparations to be immersed in cleaning solution.

6.2 *Beakers*—~~glass beakers, 50,~~ Glass beakers of (50, 100, and ~~500 ml~~ 500) ml capacity, one each for each etching setup.

6.3 *Stirring ~~Rods—~~Rods,* glass, approximately 20 cm long.

6.4 *Hot ~~Plate—~~Plate,* electric or gas-heated with capability for temperature control and rotating stirring magnets.

6.5 *Watch ~~Glasses—~~Glasses,* glass, ~~100–200~~ 100 mm to 200 mm in diameter, depending on size of specimen blocks to be etched.

6.6 *Graduated ~~Cylinders—~~Cylinders,* glass, ~~25~~(25 and ~~100~~100) ml.

6.7 *Grinding and Polishing Equipment*—~~one~~One or several laps on which the coal briquets or blocks can be ground and polished to a flat, scratch-free surface. Laps may be made of aluminum, iron, brass, or bronze.

## 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the quality of the etch.

7.2 *Potassium Permanganate* ($KMnO_4$), crystals.

7.3 *Sodium Sulfite* ($Na_2SO_3$), anhydrous, granular.

7.4 *Sulfuric Acid* ($H_2SO_4$), 47 % $H_2SO_4$.

7.5 *Sodium Hydroxide* solution, dissolve 10 g NaOH crystals in 90 g deionized water at room temperature.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

## 8. Materials

8.1 *Grinding Abrasives*—Water-resistant, adhesive-backed silicon carbide papers of ~~45,~~(45, 32, and ~~15~~15) µm ~~(240, 400,~~(240 grit, 400 grit, and 600 grit). Two or more of these can be used according to a plan such as one of those listed in Table 1.

8.2 *Polishing Abrasives*—Levigated aluminum oxide powders of 1.0 µm size (aqueous suspension) and colloidal silica of 0.06 µm size (in a prepared NaOH suspension).

8.3 *Lap Coverings*—Chemotextile material backed with water-resistant adhesive or similar quality lap coverings. Recommendations of the manufacturer of the polishing abrasive used should be followed for choice of lap covering.

8.4 *Diamond Impregnated Lap Wheel*—Impregnated with diamonds of 6 µm size.

8.5 *Detergent or Sonic Cleaning Solution*—Any nonoxidizing detergent may be used for cleaning sample surfaces after each grinding and polishing stage.

8.6 *Binder*—A potting epoxy resin and hardener or potting polyester resin and hardener that has a curing temperature less than 100 °C.

8.7 *Sample Molds*—Prepared for block samples and ~~is~~ made from potting-type silicone rubber.

8.8 *Release Agent*—Spray silicon or any other preparation that does not damage the molds or adversely affect the coal or mounting

**TABLE 1 Suggested Plans for Grinding and Polishing of Briquets and Blocks**

| Plan No. | Grinding with Silicon Carbide Paper | | | Polishing | |
|---|---|---|---|---|---|
| | Stage 1 | Stage 2 | Stage 3 | Stage 1 | Stage 2 |
| ~~1~~ | ~~45µ m (240 grit)~~ | ~~22 µm (400 grit)~~ | ~~15 µm (600 grit)~~ | ~~1 µm Alumina~~ | ~~0.06 µm~~ |
| 1 | 45 µm (240 grit) | 22 µm (400 grit) | 15 µm (600 grit) | 1 µm Alumina | 0.06 µm Colloidal Silica |
| 2 | 22 µm (400 grit) | 15 µm (600 grit) | ... | 1 µm Alumina | 0.06 µm Colloidal Silica |

medium may be used to coat the inside of the briquette mold and facilitate ejection of the briquet.

NOTE 1—Molds prepared from silicone rubber as described in Appendix X1 do not require release agent.

## 9. Sample Preparation

9.1 *Coal Briquets:*

9.1.1 Prepare granular samples as briquets in accordance with Practice D2797.

9.2 *Coal Blocks:*

9.2.1 Obtain specimens from core or as blocks of coal from a mining face.

9.2.2 Trim specimens to about 0.5 mm smaller than the volume of the silicone rubber molds.

9.2.3 Air dry the specimens to remove visible surface moisture.

NOTE 2—Overdrying specimens of low rank coals at any point in preparation can cause slaking or severe desiccation of specimen. In contrast, underdrying

of specimens will prevent epoxy from setting properly.

9.2.4 Mix resin and hardener according to manufacturer's instructions.

9.2.5 Place specimens and labels into silicone rubber molds and pour resin mixture over the specimens and labels up to the level of the top of the molds. Allow to cure, then remove the specimens from the molds.

## 10. Preparation of Sample Surface

10.1 Grind and polish on the base surfaces of the briquet or block on a lap in a wet slurry to obtain a surface suitable for microscopical examination. Grinding and polishing should be done with automated equipment. Use a series of abrasives of decreasing particle size according to a plan such as one of those described in Table 1.

## 11. Determination of Etching Time

11.1 In this procedure, the etching time is determined from the relationship between optimum etching time and measured reflectance of unetched polished vitrinite (Fig. 1).

11.1.1 Measure reflectance of vitrinite on a subsplit in accordance with Test Method D2798.

11.1.2 Using the relationship shown in Fig. 1, determine the etching time required for obtaining an optimum etch of the polished sample surface.

11.1.3 Etching time determined from vitrinite reflectance can be further optimized for lithotype for coals with a reflectance greater than 0.8 % (subbituminous) (see Terminology D121 and Table 2).

11.1.4 Samples with a vitrinite reflectance between 2.0 % and 3.5 % should be boiled within the etching solution beaker for an additional 10 s per 0.1 % greater reflectance than the etching time demonstrated in Fig. 1.

11.1.5 Samples with vitrinite reflectance greater than 3.5 % should be treated with a solution containing an additional 2.5 ml of acid for every batch solution (following the procedure outlined in Section 12, below) at an additional 10 s per 0.1 % greater reflectance than the etching time demonstrated in Fig. 1.

## 12. Etching Procedure[4]



12.1 Preparation of etching solution: To 100 ml deionized water, add and mix 25 g $KMnO_4$, and 5 ml $H_2SO_4$ (concentrated). **Caution:** always add acid to water.

12.2 Preparation of rinsing solution: To 100 ml deionized water, add 25 g $Na_2SO_3$ and 5 ml $H_2SO_4$. Stir solution until all $Na_2SO_3$ has dissolved.

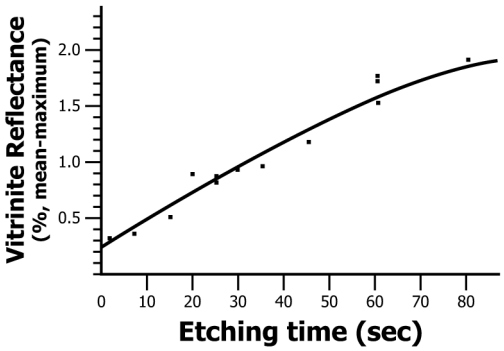


**FIG. 1 Determination of Etching Time for Coal on the Basis of Measured Vitrinite Reflectance**

[4] Modified from procedure outlined in Stach, ~~Ernst, 1935,~~ E., *Lehrbuch der Kohlenpetrographie*: Berlin, Borntraeger, 1935, 293 p.; Teichmüller, M. L., ~~1941,~~*The fine structures of American coals in polished samples and thin sections: Reichsamt für das Jahr 1940, Band 61*, ~~p.~~ 1941, pp. 20–55.

**TABLE 2 Time Adjustments Supplementary to Etching Time for Coal on the Basis of Measured Vitrinite Reflectance**

| Coal lithotype | Time adjustment(s) |
|---|---|
| Bright | + 3.5 |
| Bright Banded | + 2.5 |
| Dull Banded | - 3.5 |
| Dull | - 5.5 |

12.3 Heat the etching solution in a water bath until most of the $KMnO_4$ has dissolved.

12.4 Pour part of the heated solution into a watch glass and immediately submerse the polished coal surface in the etching solution for the time as determined from Fig. 1 and in accordance with 11.1. For samples with vitrinite reflectance greater than 2.0 %, the watch glass should remain heated to prevent recrystallization of the solution during etching.

12.5 Remove the coal surface from the solution and immediately rinse with flowing deionized water for ~~2–3~~ 2 s to 3 s to remove etching solution.

12.6 Submerse the coal surface into the rinsing solution for ~~one min~~ 1 min or until all ~~purplish~~ stain has been removed.

12.7 Clean the coal surface ultrasonically in deionized water ~~for one minute.~~or rinsing solution, for 3 min.

12.8 Dry the surface with a stream of compressed air immediately after removing from ultrasonic bath.



NOTE 3—For some samples, a small area of the polished surface can be masked using ~~cellophane~~drafting tape smoothed so as to prevent any etching effects. After etching, this tape is removed, which produces a line that demarcates the etched and unetched areas. Drafting tape is recommended, as this does not leave adhesive residues on the sample surface.

NOTE 4—For block samples of low rank coal, primarily less than 0.5 % reflectance, blocks can be stored in a shallow bath of deionized water to prevent slaking or severe drying. Prior to examination, the sample surface can be dried with a stream of compressed air. Some low rank coals may also require using a diluted etching solution for the time shown in Fig. 1.



## 13. Recognition of Common Coal Phyterals

13.1 Phyterals in etched blocks can be quantified using a similar method to standard maceral counting; a template "counting sheet" for this practice is provided in Table 3, and counted along a single transect of the sample, using a 20X air objective.

13.2 Phyteral counts can be performed on etched fragments greater than 10 µm (0.0004 in.) in size by occurrence along the transect. Particles smaller than 10 µm (0.0004 in.) are considered part of the organic matrix.

13.3 The number of counts ($n$) per sample can be constrained to the number of preserved specimens within a 5 cm (1.97 in.) long by 2 cm (0.79 in.) wide cross section of coal (optimal sample area for analysis). Therefore, with poorly preserved (dull) samples, the total count potentially is very low. The cut off for counts per sample must be determined by the user, taking into account the heterogeneity and preservation of the phyterals in the sample.

13.4 Figs. 2-8 show the most common phyteral bodies recognized in coals of Palaeozoic to Cenozoic age.[5] The appearance of phyterals is diverse and changes from sample to sample and is highly dependent on age, rank, type, palaeogeographic and palaeobotanical properties.

13.4.1 *Bark Tissue*—Bark tissues of cork cells are often only observed *in situ* with wood tissues. Bark tissues are typically first to degrade and are typically poorly preserved in vitrinite bands. Very rarely bark endoderm (cuticle) can be observed *in situ*.

[5] Van de Wetering, N., Esterle, J., and Baublys, K., "Decoupling$\delta$13C response to palaeoflora cycles and climatic variation in coal: A case study from the Late Permian Bowen Basin, Queensland, Australia," *Palaeogeography, Palaeoclimatology, Palaeoecology*, 386, 2013, pp. 165-179.

**TABLE 3 Template for Phyteral Count, [1] 'Stringy' Matrix Differentiated by Degraded Matter with Poor Cell Structure Preservation, Fine-grained with Inclusions of Dispersed Plant Tissue Longer than 100 µm, [2] 'Blebby' Matrix Material Differentiated by Degraded Matter with Poor Cell Structure Preservation, Fine-grained with Inclusions of Dispersed Plant Tissue Less than 100 µm (0.004 in.) in Length, with Spherical, Sub-spherical, Equant, or Semi-equant Dimensions**

| Phyteral Group | Phyteral Class |
|---|---|
| preserved vitrinite tissue (wood) | *determined by age, palaeogeography, and palaeobotanical properties* |
| cuticle | cuticle (other)<br>cuticle (leaf)<br>cuticle (bark)<br>cuticle (root) |
| preserved vitrinite tissues (excluding wood) | bark tissue<br>root tissue<br>interior leaf tissue (paren-/aerenchyma)<br>shoot tissue |
| megaspores | spore (ornamented)<br>spore (smooth) |
| sclerota | fungi sclerota (spore)<br>fungi sclerota (other)<br>Plantae sclerota (secretinite) |
| unpreserved tissues | degraded tissue (woody)<br>degraded tissue (fungal replacement)<br>degraded tissue (charcoal) |
| matrix | matrix (stringy)[1]<br>matrix (blebby)[2] |



13.4.2 *Cuticle Tissues*—Cuticle tissues are defined by the presence of ray cells. Cuticle from leaves (which do not preserve stomata) and roots are often indistinguishable as their anatomy is preserved similarly. Leaf parenchymal and aerenchymal tissues are rarely preserved, appearing as lens-shaped vascular bundles with some preserved mesophyll. These phyterals are often liptinite and thin vitrinite macerals.

13.4.3 *Fungal Tissues*—Fungally-degraded tissues often appear as unremarkable fusinites and semifusinites when unetched. When etched, these tissues still retain some xylem/cork structure, though most has been replaced by fungal tissue. Fungally-degraded woods often have sclerota, hyphae, cellular wall thickening and damage, and irregular oxidation patterns.

13.4.4 *Matrix and Degraded Tissues*—Matrix and degraded tissues often are very thinly laminated and compacted organic matter of various origins ("stringy" matrix), or sometimes are reworked "blebby" matrix. Stringy matrix is often associated with high liptinite content, whilst blebby matrix is associated with inertodetrinite.

13.4.5 *Root Tissues*—Root tissue can be recognized by its vascular cambium (internal ring), distinguishing it from typical shoot tissues. Typically, the distinction between these phyterals is based on the *in situ* location, that is reworked; vertically stratified tissues are defined as roots.

13.4.6 *Shoot Tissues*—Distinctions between root and shoot tissues in etched coal are often unclear; degradation of the outer cuticle and reworking of peat mask small cellular differences between these phyterals. Shoot tissues, though rarely preserved, are horizontally-stratified, and often show remnant imbricate structure, stipules, and petioles.

13.4.7 *Xylem Tissues (Wood)*—Wood tissues are defined by the presence of recognizable xylem. Common wood tissues have either pycnoxylic (low parenchyma tissues between xylem) or manoxylic (abundant parenchyma tissues between xylem). The appearance of preserved wood tissues varies on taxonomy, preservation, compaction, and cross-section view. In coals cut to view stratification, the most common view is cross-sectional or tangential.

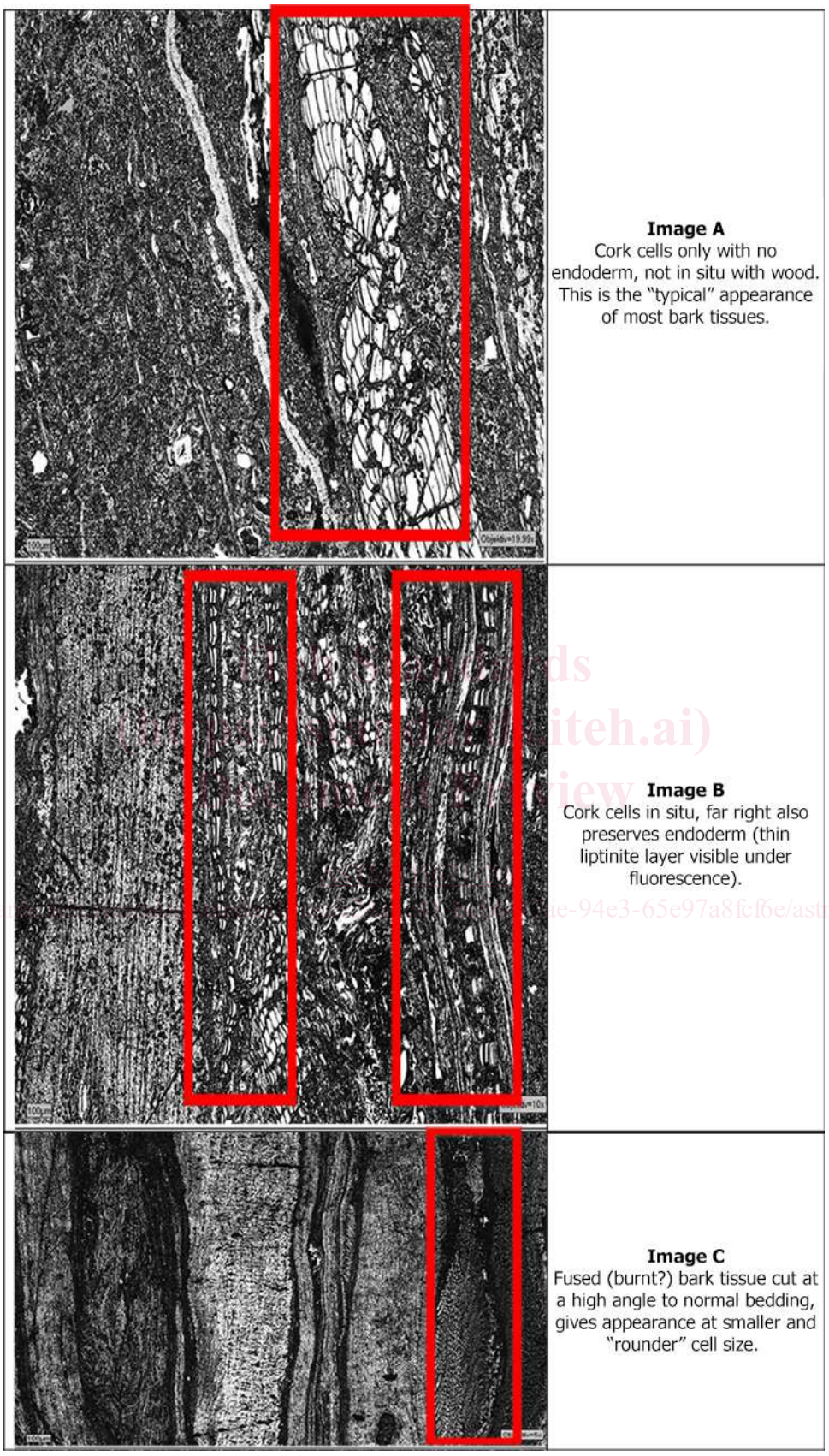


**FIG. 2 Examples of Bark Phyteral Occurrence**